# $^{13}$C NMR STUDY OF ALBENOSCHUS ESCULENTUS COMPOUNDS

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**Keywords**: $^{13}$C solution NMR, solid state NMR, Albenoschus Esculentus

## Abstract

The investigation of the main compounds presented in the *Albenoschus Esculentus* (AE) has been carried out employing nuclear magnetic resonance spectroscopy (NMR), using solution and/or solid state techniques when it one was necessary. The evaluation of NMR data allowed us to characterize the main type of components presented in this kind of sample. It was necessary to use a total information from solid state NMR and also the solution response. From these informations we could get that four main components were presented in this sample. One in the shell, that is cellulose; another one between the shell and seeds that is a polysaccharide and in the seed two components were found one is a starch and the second one is an oil, a triacylglycerol. These components are responsible by its physical chemistry properties.

## Resumo

A investigação dos componentes principais presentes no the *Albenoschus Esculentus* (AE) vem sendo realizada pelo emprego da espectroscopia de ressonância magnética nuclear (RMN), usando técnicas em solução e/ou estado sólido quando necessárias. A avaliação dos dados de RMN permitiram caracterizar os principais tipos de componentes presentes neste tipo de amostra. Foi necessário o uso das informações advindas dos resultados da RMN do estado sólido assim como das respostas de solução. A partir destas informações foi possível detectar quatro componentes principais estão presentes nesta amostra. Um está localizado na casca, que é a celulose; outros entre a casca e a semente, que é um polissacarídeo e na semente dois componentes foram encontrados um é um amido e o outro um óleo, um triacilglicerídeo. Estes componentes são responsáveis pela propriedades físico-químicas do AE.

## Introduction

The investigation of the main compounds presented in the *Albenoschus Esculentus* is necessary to better understand its application in water treatment as a clarify agent. Among the many experimental techniques that can be employed to study chemical structures, solution and solid state nuclear magnetic resonance have proved to be particularly successful. It is known that solution techniques provide detailed information on chemical structure and microstructure. The solid state NMR experiments also provide information on chemical structure without interfere in the sample, because NMR is a non-destructive spectroscopy[1-5]. Therefore, data on the molecular dynamics can be also obtained by solid state NMR. Thus, the use of both solution and solid state NMR many responses can be extracted for the materials[1-9].

The main purpose of this work is to obtain information on the main chemical components of *Albenoschus Esculentus* to better understand its behavior when it is use in water treatment. To obtain the responses for our purpose we have chosen to characterize all fruit by solution and solid state NMR techniques.

**Experimental**

The methodology of analysis chosen is described bellow.

***Albenoschus Esculentus***

Two types of sample treatments were done:

1 – The AE vegetable was dried and powdered, after that the extraction of the polysaccharide was carried out using different solvents:

1.1 – water, deuterated acetone, deuterated dimethylsulfoxide, $KOH/D_2O$ and deuterium oxide

1.1.1 - The extracted were first analysed by $^{13}C$ solution NMR analysis and after that a $^{13}C$ solid state NMR analysis was done

2 – The AE vegetable was dried and the seed isolated

$\Rightarrow$ 2.1 - the shell was powdered

$\Rightarrow$ 2.2 - the seed was powdered and an extraction of the oil by solvent (acetone) was done

$\Rightarrow$ 2.3 - and a $^{13}C$ solution NMR analysis was carried out

All NMR solution spectra were carried out on a VARIAN MERCURY 300 and the solid state experiments were obtained on a VARIAN INOVA 300. Both spectrometer operating at a $^{13}C$ resonance frequency of 75.4 MHz. The $^{13}C$ solution spectra were obtained in adequate quantitative conditions. All solid state were recorded with magic angle spinning (MAS), using a short delay between 90 degree pulses. The cross-polarization magic angle spinning (CPMAS), had a contact time of 1 ms and the cross-polarization magic angle spinning with dipolar dephasing (CPMASDD), used the same conditions as was established for CPMAS with a dephasing time 40 $\mu$s.

**Results and Discussion**

According to the methodology described before, the dried and powder AE was analyzed by $^{13}C$ NMR solution in different solvents to detect the polymeric component, probably a polysaccharide, that can have properties to be applied in water treatment. The solubilization was carried out in deuterated acetone $(CD_3)_2CO$, deuterated dimethylsulfoxide DMSO, pair of $KOH/D_2O$ and deuterium oxide. All extracted were analysed and the interpretation of those spectra showed that: the extracted obtained in $(CD_3)_2CO$ only showed signals that belongs to the aliphatic region, those signals were attributed to an oil, probably, a triacylglycerol that derives from the seed. The $^{13}C$ spectrum obtained from the extracted in DMSO shows signals from polysaccharide, located at 99 ppm (C-O anomeric); 68-82 ppm (CH-O) and 62 ppm ($CH_2$-O) and also from cellulose located at 104 ppm (C-O anomeric); 84 ppm (CH-O) and 60 ppm ($CH_2$-O) (Figure 1). The $^{13}C$ spectrum of the extracted obtained by $KOH/D_2O$ showed the same signals detected by DMSO. The water solution did not show any signal.

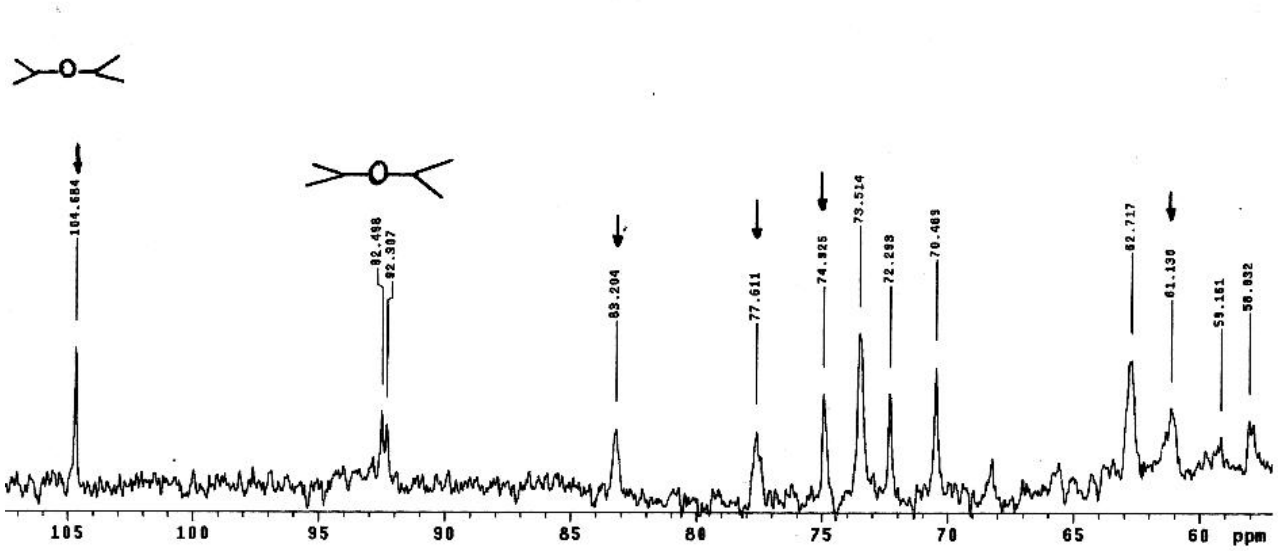


Figure 1 - The $^{13}C$ spectrum obtained from the extracted in DMSO

The $^{13}C$ solid state spectra were recorded using a CPMAS and MAS techniques. Signals from polysaccharide, cellulose and oil were detected.

After seed isolation, an oil extracted from it was characterized by $^{13}C$ solution NMR using $(CD_3)_2CO$ as a solvent. All signals detected were attributed to a triacylglycerol, those signals were the same found in the solid state NMR (Figure 2).

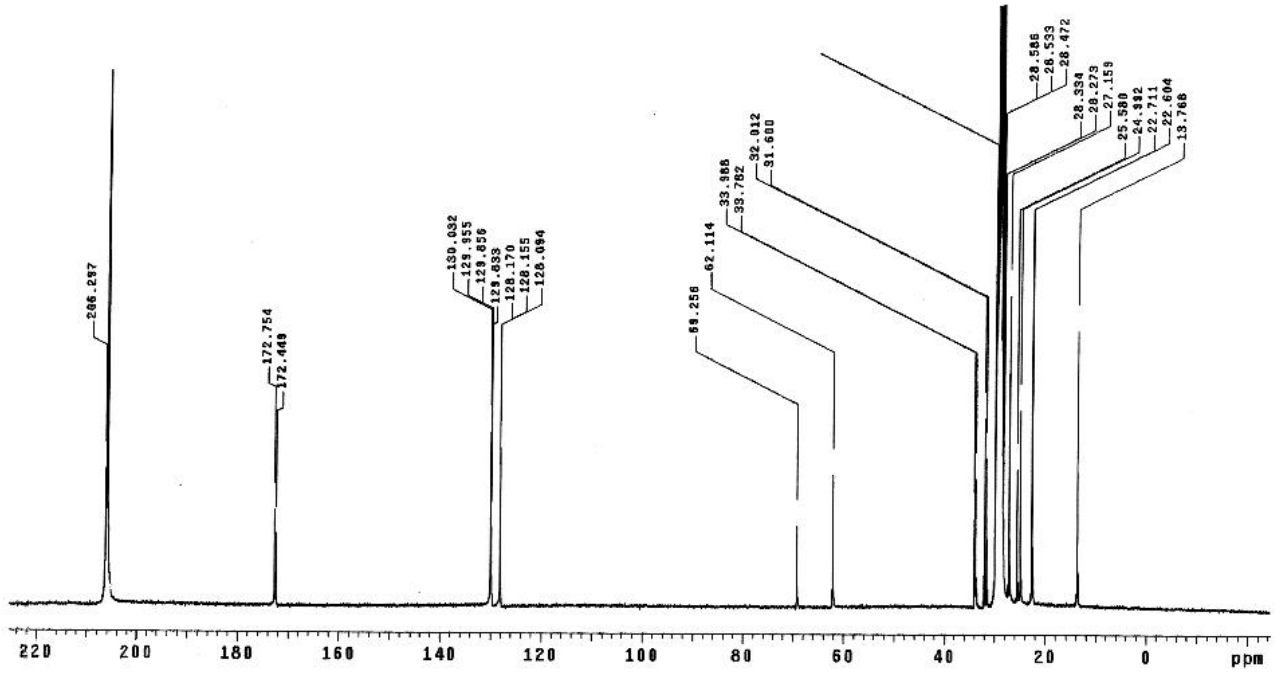


Figure 2 - $^{13}C$ solution NMR of an oil extracted from AE, using $(CD_3)_2CO$ as a solvent

The CPMAS $^{13}C$ solid state NMR study of the seed flour showed that the main component that is, probably, a starch due to the detection of signals related to a mono and di and polysaccharides (Figure 3). MAS and CPMASDD $^{13}C$ techniques were also carried out and the results obtained confirmed the data characterized by the analysis of the whole AE powder.

From Figure 3 it is clear that the CPMAS $^{13}C$ main signals assigned in the spectrum were derived from the starch and weak lines located at about 34 and 145 ppm were probably attributed to the gluten proteins.

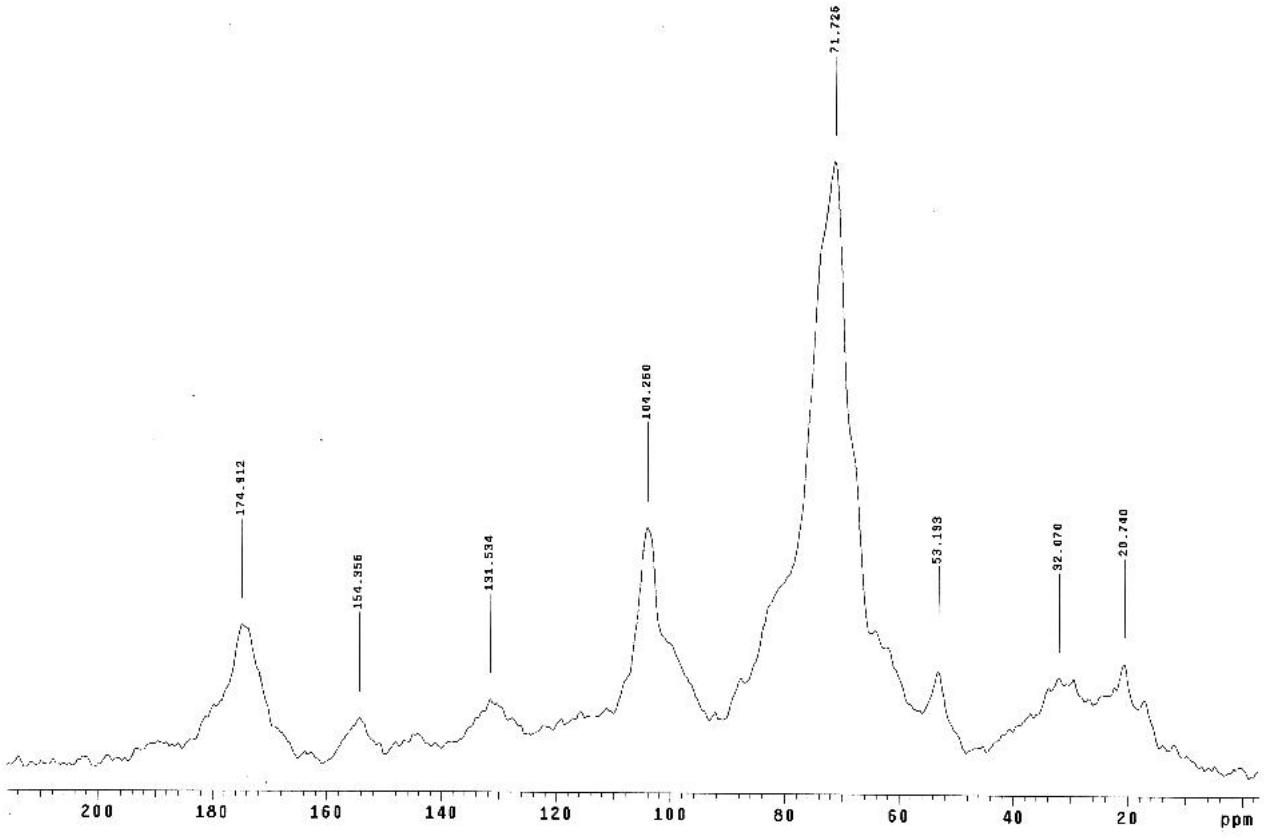


**Figure 3** - CPMAS $^{13}C$ solid state NMR spectrum of AE seed starch.

## Conclusions

According to the main purpose of this work, both solution and solid state NMR techniques provided valuable informations on the main chemical components presented in the AE without any chemical treatment. The response of the AE components, which was monitored by MAS, CPMAS, CPMASDD showed that the molecular mobility is influenced directly from the mix of the components presented in the material, causing a sample heterogeneity.

## Acknowledgements

The authors would like to thank you PRONEX-CNPq 0327.00/00 and FAPEMAT, for the financial support. And to PETROBRAS/CENPES/Gerência de Química for the use of solid state NMR spectrometer.

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